Union of Soviet Socialist Republics	DESCRIPTION OF AN INVENTION for a Certificate of Authorship	(11) <b>644731</b>
	(61) Addition to Certificate of Authorship — (22) Filed on 5/24/74 (21) 2027678/23-26	
State Committee of the USSR for Inventions and Discoveries	joining with application No. —  (23) Priority —	(51) Int. CI. <sup>2</sup> C 01 B 33/24
	(43) Published <b>1/30/79</b> , Byulleten' No. 4 (45) Date specification was published 1/30/79	(53) UDC 661.183.6 (088.8)
(72) Authors of the invention:	M. G. Manvelyan and G. G. Martirosyan	
(71) Applicant:	Institute of General and Inorganic Chemistry, Armenian Academy of Sciences	

## (54) METHOD FOR PREPARING SYNTHETIC DIOPSIDE

The invention relates to the technology for preparing synthetic diopside, used in the production of ceramic products, polymers, paints, paper, and resins.

A method is known for preparing synthetic diopside by the joint calcination of silicic acid with previously ground calcium and magnesium-containing products, for example, oxides of calcium and magnesium, magnesite, limestone, or dolomite. The calcination process is carried out at 1260–1390 °C [1].

A drawback of the known method is the necessity of using high temperatures during calcination and the low yield of the target product, not exceeding 80%.

A method for the preparation of synthetic diopside is known, according to which calcium-magnesium hydrosilicate, obtained by the hydrothermal treatment of a mixture of ground dolomite or oxides of calcium and magnesium with quartz sand at a temperature above 170°C, is calcined at 1200–1250°C. In this case, the possibility of preparing the target product by this method with a diopside yield of not less than 95% is achieved only with a calcination time on the order of 10 h. As a result, a diopside with a specific surface up to 6.0 m²/g is obtained [2].

Nevertheless, this method even with several hours of calcination does not make it possible to obtain a product with a diopside yield of more than 97%. With calcination for an hour, the product has a yield of only 80% and a specific surface not exceeding 2 5–4 0  $\text{m}^2/\text{g}$ .

The object of the invention is to increase the yield and the dispersion of the target product and to intensify the process for its preparation.

This object is achieved by the described method for obtaining synthetic diopside by calcination of the product, obtained by caustification of alkaline silicic solutions with calcined dolomite having an alkali content of 0.5–1.0% by weight, in terms of sodium oxide, at 900–1100°C.

The distinguishing feature of this method is that the indicated product of the caustification of the alkaline silicic solutions with calcined dolomite is used as the starting raw material; in this case, the calcination is carried out at the aforementioned temperatures.

The described caustification product is obtained by hydrothermal treatment at 70–90°C for 40-90 min of the calcined dolomite and alkaline silicic solution, obtained during the alkaline hydrothermal treatment of silicon-containing rocks, for example, nepheline syenite or perlite, with a subsequent washing of the obtained caustification products to an alkali content of 0.5–1.0% by weight, based on Na<sub>2</sub>O.

The proposed method achieves an increase in the yield of the target product, in terms of diopside, up to 98% and in its dispersion to 6.0–11.5 m<sup>2</sup>/g with considerable intensification of the process resulting from shortening the process to 0.5–1 h.

Example 1. 10 L of the alkaline silicic solution, containing 80 g/L of  $Na_2O + K_2O$  and 60 g/L of  $SiO_2$ , is heated in a reactor to 80°C; 500 g of calcined dolomite containing 87% CaO and MgO is added; the mixture is mixed for an hour and filtered. The forming precipitate is washed with warm water to a content of 0.5% by weight of  $Na_2O_1$  and then calcined at 1000°C for an hour.

The obtained product contains 98% diopside and has a specific surface of 10 m<sup>2</sup>/g. The distribution by percent of the particles in microns is as follows:  $100-40 \mu$ , 15%;  $40-10 \mu$ , 75%;  $10-1 \mu$ , 9.0%; and  $1 \mu$ , 1.0%.

Example 2. The precipitate of calcium-magnesium hydrosilicate, obtained as in Example 1 by caustification of the alkaline silicic solution, is washed to a Na<sub>2</sub>O content of 1 0% by weight, and then calcined at 900°C for an hour

A product is obtained with a 98% diopside content and with a specific surface of  $11.5 \text{ m}^2/\text{g}$ .

The method for preparing synthetic diopside as taught by the invention is sufficiently economical, because it permits a decrease in energy expenditures owing to a decrease in the calcination temperature and owing to the calcination productivity.

In addition, the increased dispersion and higher diopside content in the target product improves its quality and broadens the possibility of its use in various materials.

## Claim

Method for the preparation of synthetic diopside, comprising the calcination of a calcium-magnesium hydrometasilicate, characterized in that to increase the yield and dispersion of the target product, and also to intensify the process, the product of the caustification of alkaline silicic solutions with calcined dolomite having an alkali content of 0.5–1.0% by weight, in terms of sodium oxide, is used as the starting raw material, and calcination is performed at 900–1100°C.

References considered in the examination

- 1. West German patent No. 1959662, class C 01 B 33/24, 7/3/71.
- 2. English patent No. 1272042, class C 1 A, 4/26/72.